GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station

PROJECT INITIATION

Date: November 15, 1974

Project Title: Evaluation of Shell Molding Techniques as a Means of Forming Precision Cast Silicon Carbide Shapes

Project No.: A-1692

Project Director: Mr. J. N. Harris

Sponsor: Ford Motor Co. (Turbine Research Dept.); Dearborn, Michigan

Effective November 8, 1974 Estimated to run until March 7, 1975

Type Agreement: Std. Industrial dtd 9/20/74 ~~Ford's P.O. 47K-375926-LB~~ Amount: $ 20,000

Reports Required: Monthly Technical Letters; Final Technical Report

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# SPONSORED PROJECT TERMINATION

Date: 7/21/78

Project Title: Evaluation of Shell Molding Techniques as a Means of Forming Precision Cast Silicon Carbide Shapes

Project No: A-1692

Project Director: Mr. J. N. Harris

Sponsor: Ford Motor Co. (Turbine Res. Dept.) Dearborn, Michigan

Effective Termination Date: 9/30/75

Clearance of Accounting Charges: All Cleared

Grant/Contract Closeout Actions Remaining:

NONE

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- _ Final Report of Inventions
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ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

December 11, 1974

Ford Motor Company
Turbine Research Department
2000 Rotunda Drive
Scientific Laboratory
Room E-3172
Dearborn, Michigan 48121

Attention: Mr. J. Secord

Subject: Technical Letter Report No. 1 for the Period 8 November-30 November 1974, P. O. No. 47K-375926-LB, "Evaluation of Shell Molding Techniques as a Means of Forming Precision Cast Silicon Carbide Shapes," Georgia Tech Project A-1692

Gentlemen:

1. Development of Shell Molds

It is anticipated that in the early stages of this work the techniques developed for forming ceramic shell molds under P. O. No. 39K-107938-LB will be followed very closely except the mold material will be carbon or graphite. Granular forms of carbon and/or graphite are not as readily available in the Georgia Tech area as were the ceramic materials used in the previous work. Therefore, it has been necessary to order these materials. Materials have been ordered in mesh sizes of: 6 to 14, 50 to 200, -100 and -325.

In order to begin studies on the formation of carbon molds an available graphite tube was ground and screened to produce materials of -200 and -325 mesh. These fractions were blended to approximate the P-1 and P-2 powder sizes listed in the NALCO product literature for fused silica. The powders were slurried with a 10 weight percent Polyvinyalcohol solution to act as a binder. Compositions of the two slurries are shown in Table I.

Single and multi-blade wax patterns were dipped in the slurries and then coated by sprinkling with a -50 mesh graphite stucco. No problems were encountered with wetting of the wax. After drying the wax was removed by placing the molds in a 100° C dryer until the majority of wax had melted out. None of the molds cracked due to thermal expansion of the wax. The molds were then washed in toluene to remove the remaining wax and finally washed in acetones to remove the toluene. If the toluene was not washed out the PVA bond was weakened and the mold collapsed.

TABLE I

SLURRY COMPOSITION USED TO FORM GRAPHITE MOLDS

1st and 2nd Coats

| Material | Weight Percent |
|---|---|
| 10 w/o PVA solution | 69 |
| Graphite Powder 27% -200 +325 | 31 |

3rd and 4th Coats

| Material | Weight Percent |
|---|---|
| 10 w/o PVA solution | 69 |
| Graphite Powder 84% -100 +200 | 31 |
| 15% -200 +325 | |
| 1% -325 | |

## 2. Development of Carbon Mold Materials

Studies were conducted to produce granular carbon mold materials by carbonization of various resins and other organic materials. The resins were filled with lampblack before curing in efforts to increase the yield. Carbonization was accomplished in a tube furnace by heating to 1000° C in a nitrogen atmosphere. Granular carbon materials were formed in this manner, however, the yield was only 14 percent with epoxy resins. Phenolics are expected to give a better yield, but the phenolic resins were not received in time for inclusion of the data in this report.

## 3. Anticipated Problem Areas and Future Work

At this time no major problems are anticipated. During the next report period work will continue on developing carbon molds and materials and on improving the techniques of wax removal. Initially molds will be filled

with silica slips and fired in an inert atmosphere to determine ease of mold removal. If no problems occur in this operation then molds will be filled with the silicon carbide slip and carried through the complete sintering operation.

Respectfully submitted,

Joe N. Harris
Project Director

jw

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

January 14, 1975

Ford Motor Company
Turbine Research Department
2000 Rotunda Drive
Scientific Laboratory Building
Room E-3172
Dearborn, Michigan 48121

Attention: Mr. J. Secord

Subject: Technical Letter Report No. 2 for the Period 1 December-31 December 1974, P. O. No. 47K-375926-LB, "Evaluation of Shell Molding Techniques as a Means of Forming Precision Cast Silicon Carbide Shapes," Georgia Tech Project A-1692

Gentlemen:

1. Development of Carbon Mold Materials

Attempts were continued to obtain granular carbon particles for slurries and mold stucco by carbonization of organic materials. Blending 42 weight percent lampblack into the epoxy resin before curing resulted in an increase in yield to 46 percent upon carbonization in nitrogen at 1900° F. However, the product obtained was softer and more friable than that obtained by carbonizing the epoxy resin alone. Phenolic resins have been obtained and will be used in an effort to increase the yield of carbon particles.

2. Development of Carbon Shell Molds

Full rotor molds were formed using the carbon slurry. These molds were dewaxed by placing in a dryer at 100° C until the majority of the wax had melted out. Additional wax was removed by washing with toulene and acetone. No problems of mold cracking have occurred with all carbon molds due to thermal expansion of the wax during melting.

3. Future Work

Due to the limited availability of the silicon carbide powder for slip casting and the short work month, the silicon carbide slip was not made during December. It is anticipated that the slip will be formed and carbon molds filled for sintering during January.

Respectfully submitted,

Joe N. Harris
Project Director

jw

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

February 11, 1975

Ford Motor Company
Turbine Research Department
2000 Rotunda Drive
Scientific Laboratory Building
Room E-3172
Dearborn, Michigan 48121

Attention: Mr. J. Secord

Subject: Technical Letter Report No. 3 for the Period 1 January-31 January 1975, P. O. No. 47K-375926-LB, "Evaluation of Shell Molding Techniques as a Means of Forming Precision Cast Silicon Carbide Shapes," Georgia Tech Project A-1692

Gentlemen:

1. Development of Carbon Mold Materials

Due to the time required no attempts to form carbon materials from resin chars was made during this report period. Commercially available materials were used instead. Slurries were made using a ten percent aqueous polyvinyl alcohol solution and -325 mesh electrode grade carbon from the Ultra Carbon Corporation. Stucco materials were prepared by grinding and screening "HC" grade graphite rods.

2. Carbon Shell Mold Development

Carbon shell molds were fabricated in the form of three blade sections and full rotors both for this program and P. O. No. 39K-107938-LB. Because of the shortage of carbon stucco materials some of the molds were stuccoed with silicon carbide grain.

Two techniques of dewaxing were investigated. The first technique consisted of "cutting-out" the majority of the wax from the mold using a hot soldering iron then placing the mold with the remaining wax in a 100° C oven. After allowing sufficient time for the wax to melt and run out, the molds were removed from the oven and cooled. The remaining wax film was removed by filling the mold with warm (65° C) toluene. Upon removal of the toluene the molds were rinsed with acetone at room temperature.

The second method consisted of heating the mold with wax pattern intact with microwave energy. This caused initial melting of the wax at the

mold-wax interface. The large wax cores fell out of the molds without melting. After removing the majority of wax the toluene and acetone treatment previously described was used to completely remove the last traces of wax.

3. Silicon Carbide Slip

All materials required to mill the silicon carbide material into slip are on hand with the exception of tungsten carbide balls. These have been ordered.

4. Vacuum Firing

The vacuum furnace to be used in sintering the silicon carbide parts has been checked out in a test firing of rare earth parts. The metallic element may require replacement with a carbon element, however, the first firing will be attempted with a metallic element.

5. Future Work

During the next report period the silicon carbide slip will be prepared and vacuum sintering of parts cast in the carbon molds will be conducted.

Respectfully submitted,

Joe N. Harris
Project Director

jw

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

March 17, 1975

Ford Motor Company
Turbine Research Department
2000 Rotunda Drive
Scientific Laboratory Building
Room E-3172
Dearborn, Michigan 48121

Attention: Mr. J. Secord

Subject: Technical Letter Report No. 4 for the Period 1 February-28 February 1975, P. O. No. 47K-375926-LB, "Evaluation of Shell Molding Techniques as a Means of Forming Precision Cast Silicon Carbide Shapes," Georgia Tech Project A-1692

Gentlemen:

## 1. Carbon Mold Materials

No further attempts to produce carbon mold making materials in-house were made due to a time shortage. A commercial source of carbon powders of sufficient purity for mold making was found, Union Carbide Corporation. Union Carbide has available a wide variety of carbon powders of various sizes at economical prices. Calculations show these powders (ash content < 0.2 percent) to be cheaper than those that could be made from phenolic resins considering the weight loss that occurs during carbonization of the phenolic. These powders have not been ordered due to the remaining supply of Ultra Carbon UCP-1-325.

## 2. Carbon Shell Mold Development

Molds were fabricated in the form of full rotors and three blade sections to further refine the technique. Optimum carbon slurry composition was found to be 25 w/o carbon (Ultra Carbon UCP-1-325) in 75 w/o of a 10/o PVA solution. More carbon or higher PVA concentrations increase the viscosity of the slurry and too much entrained air remains to cause bubbles (holes) in the mold surface. A less viscous slurry runs quickly off the wax before stuccoing can be carried out. Wetting agents (Ultrawet) cause foaming and do not appreciably improve wetting of the wax pattern by the slurry. Anti-foaming agents (DOW DB100) cause a reduction in wetting ability. The problem of slurry wetting ability remains. Dewaxing has proceeded smoothly using both the soldering iron and microwave energy techniques described in the last monthly letter report. In an attempt

to check the surface finish of a casting made in the carbon molds, a full rotor mold was filled under vacuum with a fused silica slip. The mold will be burned off the fused silica and the surface of the casting can then be examined.

3. Silicon Carbide Slip

A small batch of silicon carbide slip was milled and used to fill two three-blade carbon molds. The filling was done by vibration without vacuum. The filled molds were allowed to dry in air at room temperature and a great deal of shrinkage was evident after drying. These castings have not yet been fired.

4. Vacuum Firing

A graphite element has been constructed for the vacuum furnace to be used to sinter the silicon carbide castings. The furnace is undergoing checks with the new element.

5. Future Work

The silicon carbide castings will be sintered in the vacuum furnace as soon as possible. Further checks on the carbon molds including a check of the inside surface as described above will be carried out. Investigation into possible workable wetting agents for the carbon slurry will proceed. The possibility of dipping the wax patterns under vacuum will also be considered.

Respectfully submitted,

Joe N. Harris
Project Director

jw

A-1692

SUMMARY REPORT

# EVALUATION OF SHELL MOLDING TECHNIQUES AS A MEANS OF FORMING PRECISION CAST SILICON CARBIDE SHAPES

PROJECT A-1692

P. O. No. 47-K-375926-LB

By

J. O. Tarter and J. N. Harris

Prepared for

FORD MOTOR COMPANY
TURBINE RESEARCH DEPARTMENT
DEARBORN, MICHIGAN 48121

July 1975

1975

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
Atlanta, Georgia 30332

SUMMARY REPORT

# EVALUATION OF SHELL MOLDING TECHNIQUES AS A MEANS OF FORMING PRECISION CAST SILICON CARBIDE SHAPES

J. O. Tarter
and
J. N. Harris

July 1975

Project A-1692
P. O. No. 47-K-375926-LB

Prepared for

Ford Motor Company
Turbine Research Department
Dearborn, Michigan 48121

Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

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# I. INTRODUCTION

The objective of this program is to develop a method for the slip casting of complex shapes of sinterable silicon carbide. The mold in which the cast silicon carbide material is contained must not interfere with the drying or sintering shrinkage of the part, but must at the same time provide sufficient support and protection so that the part maintains its integrity until some sintered strength is obtained.

The technique of using ceramic shell molds for slip casting of metal powders has been used to form experimental complex silicon nitride parts. In this procedure silicon metal power is slip-cast in the porous ceramic mold. The entire assembly is then placed in a furnace and the silicon shape is reaction sintered within the mold and under a nitrogen atmosphere to form silicon nitride. During reaction sintering the ceramic shell mold loses its strength and is separated from the final sintered part. Similar methods and techniques have been applied to forming sinterable silicon carbide shapes, but using different mold materials and sintering techniques. Organically bound carbon molds were formed and a slip-cast silicon carbide shape was sintered in one of these molds.

## II. PURPOSE

The ultimate purpose of this program was to develop a molding techniques to produce a full scale, one piece turbine rotor of sinterable silicon carbide formed by slip casting silicon carbide or using a modified slip casting technique. The primary objective for the initial work done here was to develop materials to fabricate shell molds of rotor blades and complete rotors. The feasibility of using these molds for sintering silicon carbide was also to be examined.

## III. EXPERIMENTAL PROGRAM

Carbon was chosen as the mold material because of its compatability with silicon carbide. An organic binder (polyvinyl alcohol) that would carbonize on heating in vacuum or inert gas was selected to provide green strength for the mold until firing was underway.

### A. Summary of Investment Mold Fabrication Procedure

The prepared wax pattern was dipped in a slurry consisting of 25 weight percent (w/o) -325 mesh carbon powder in 75 w/o of a 10 w/o polyvinyl alcohol solution. The wet pattern was stuccoed with -50 +140 mesh carbon powder. After drying, the dip and stucco procedure was repeated. A third and final dip and stucco were performed using the above slurry and -25 +50 mesh carbon for stuccoing. The mold was dewaxed by melting out the majority of the wax then cleaning chemically using toluene followed by acetone to remove the last traces of wax.

### B. Carbon Investment Molds

#### 1. Preparation of Wax Pattern

In order to provide a handle for dipping and/or to produce a mold shape that would produce the desired final product, the wax patterns as received were modified by "welding" additional wax parts to the patterns. Examples of these modified patterns appear in Figure 1. After modification of the turbine rotor pattern to suit the particular application, the entire pattern was cleaned to remove grease and dirt present on the wax surface. The pattern was agitated in trichloroethylene for a few seconds, rinsed in

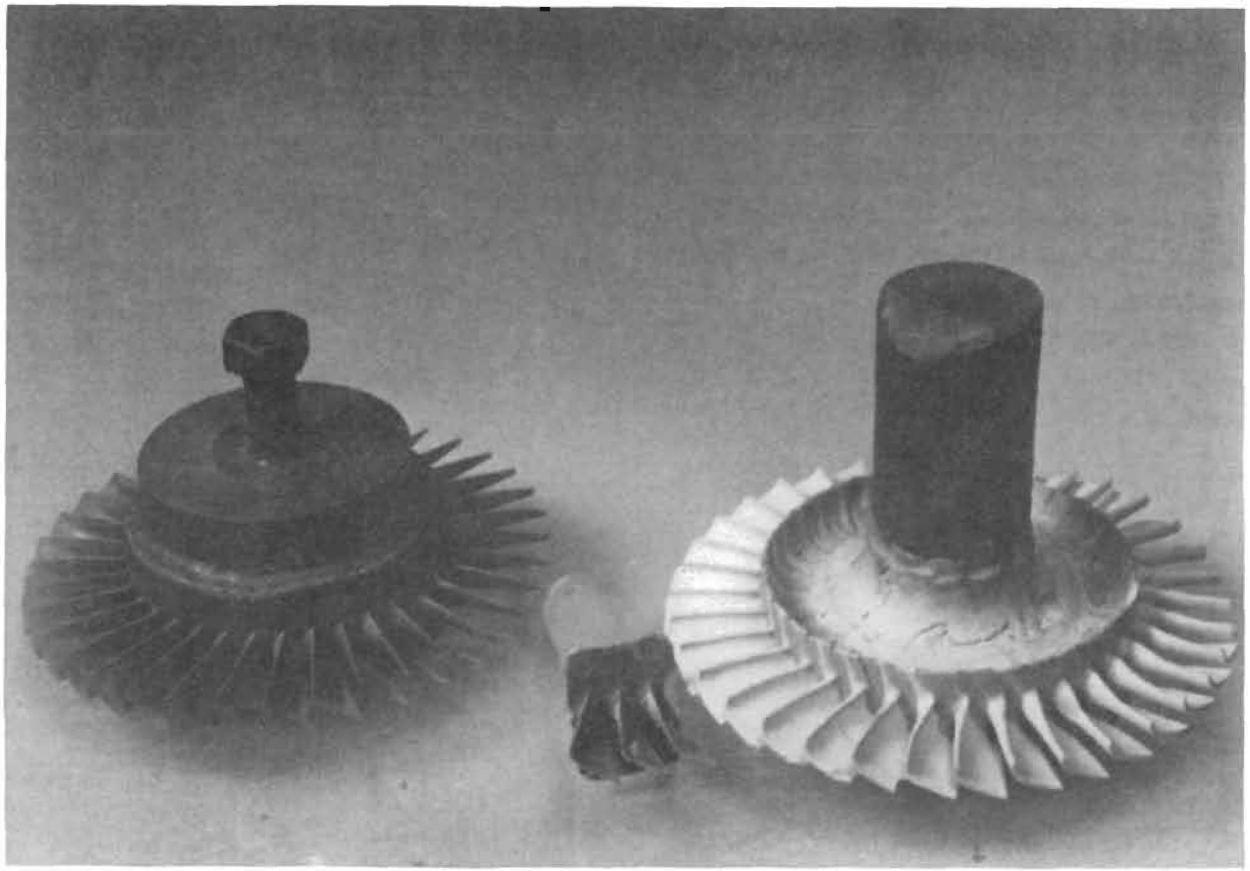

Figure 1. Modified Wax Rotor Pattern, Ready for Dipping.

isopropyl alcohol to remove the trichloroethylene, and allowed to air dry for about 45 minutes. The pattern was then ready for dipping.

2. Preparation of Polyvinyl Alcohol Solution

A mixture of 10 w/o dry polyvinyl alcohol (PVA) powder (Dupont Elvanol, fully hydrolyzed) and 90 w/o tap water (typically a 2000 gm batch) was placed in a stainless steel container and mixed with a high speed laboratory mixer (Lightnin®). A drop of Dow Corning DB100 antifoam agent was added to control foaming during the mixing operation. The mixture was heated to the boiling point and removed from the heat just as boiling started. After cooling, the solution was filtered through a coarse filter to remove the large gelatinaous masses that formed upon cooling. This solution could then be stored at room temperature indefinitely for later use. More concentrated solutions of PVA had a tendency to gel over a period of time at room temperature. Less concentrated solutions did not provide a bond of sufficient strength to withstand normal handling of the molds.

3. Preparation of Carbon Slurry

A carbon slurry was prepared by adding 25 w/o carbon powder to 75 w/o polyvinyl alcohol solution (PVA) prepared as described above. The carbon powder used was Ultra Carbon UCP-1-325 electrode grade carbon (-325 mesh) from Ultra Carbon Corporation, Bay City, Michigan.

Attempts were made to carbonize organic materials such as epoxy, phenolic resins and polyvinyl chloride to produce carbon powder for slurries and stuccoing. These procedures were time consuming and yields were low. To save time "electrode grade" carbon was purchased from Ultra Carbon. Less pure carbon could possibly have been used, especially for second and third mold layers, but the effect of the ash content, primarily silica,

on the sintering of silicon carbide was not known. Later, a cheaper source of less pure carbon from Union Carbide Corporation was obtained, but molds were not fabricated from this carbon because of the supply of Ultra Carbon Powder on hand. The Union Carbide powder (< 0.2 percent ash) is less expensive than carbon produced by carbonizing organics.

The carbon powder and PVA were mixed with a high speed laboratory mixer (Lightnin®). The carbon settled out in about 15 minutes, but could be easily redispersed with the mixer. Other percentages of PVA and carbon were tried, but the 25-75 mixture was most satisfactory. The slurry had a viscosity of 300 centipoise measured with a Brookfield Model LVF viscometer at 6 rpm. The high viscosity of the slurry tended to entrap air, but was necessary for the dipping process.

A more viscous slurry (high carbon content) entrapped a prohibitive amount of air and when dry tended to fall apart due to lack of binder. When less carbon was used, resulting in a lower viscosity, the slurry ran off the wax pattern too rapidly to allow sufficient time to apply stucco. Additional antifoaming agent did not reduce the amount of air entrapped by the viscous slurry and was harmful in that it reduced the already limited wetting ability of the slurry.

### 4. Preparation of Stucco Materials

Carbon grains for stuccoing were made by turning a graphite cylinder in a lathe to produce grains. The cylinder was an Ultra Carbon "HC" grade electrode (U644-R). Less pure and cheaper carbon or graphite grains, from Union Carbide Corporation, could have been used for stuccoing. The grains collected from the lathe were screened to separate mesh sizes

of +25; -25 +50; -50 +140; and -140 mesh. The -25 +50 mesh grains were selected for use as "coarse stucco" and the -50 +140 mesh grains were used as "fine stucco." Finer stucco powder tended to produce the same effect as additional carbon in the slurry and the mold fell apart due to a lack of binder. Coarser stucco "pulled out" of the mold during dewaxing and normal handling. The coarse stucco was used as the last coat to provide a more porous mold.

5. Build-Up of Layered Carbon Molds

The cleaned pattern was dipped into the slurry described above and agitated to dislodge large air bubbles. The slurry was stirred just before dipping with a high speed mixer. This procedure entrained air, but was necessary to disperse the carbon. It was found to be advantageous to remove the pattern from the slurry, shake off excess slurry, and redip several times until all large bubbles were broken up and a smooth layer of slurry remained on the pattern. As many as five dips were required. After the final dip the pattern was removed from the slurry, allowed to drain for about ten seconds, and stuccoed with fine grain (-50 +140 mesh). The fine grain was sprinkled onto the wet pattern from all directions until the surface just appeared to be dry. Too much stucco caused a lamination containing no binder in the finished mold which separated during subsequent dewaxing. Too little stucco allowed the slurry to run off the wax leaving a "hole" in the mold. The slurry did not actually wet the wax, but its high viscosity prevented its running off the pattern during the stuccoing operation.

Several wetting agents were added to the slurry in an attempt to promote wetting of the wax, but no water soluble wetting agent was found that would wet the wax. Solvent soluble wetting agents were not used because of their incompatability with the PVA solution.

The first coat of slurry and stucco were allowed to dry for at least five hours and in most cases overnight. If examination showed the first coat to have a hole in it, the hole was repaired by rubbing a small amount of slurry into the hole. The coated pattern was again dipped into the slurry and agitated slightly. After allowing the pattern to soak for about three seconds it was removed and allowed to drain until dripping essentially stopped; about twenty seconds. During this draining time the pattern was rotated slowly by hand to prevent an uneven layer of slurry from building up. The wet pattern was stuccoed with fine grain. After drying at least five hours the dipping procedure was repeated and stuccoing done with coarse grain (-25 +50 mesh). The coarse grain was poured on in large quantities and the pattern shaken to remove the excess. Too short a dipping time resulted in a layer of dry carbon powder not bonded with PVA causing the mold to come apart later. Too long a dipping time caused the previous layer to wash off and excessively contaminated the slurry.

The major problem encountered in the dipping procedure was the inability of the slurry to wet the wax surface, making it difficult to obtain a smooth casting surface on the molds. The hydrophobic character of the wax is illustrated in Figure 2. Entrapped air in the slurry compounded the surface problem. For this reason the slurry was put under vacuum before dipping to remove air bubbles. The dipping reintroduced air

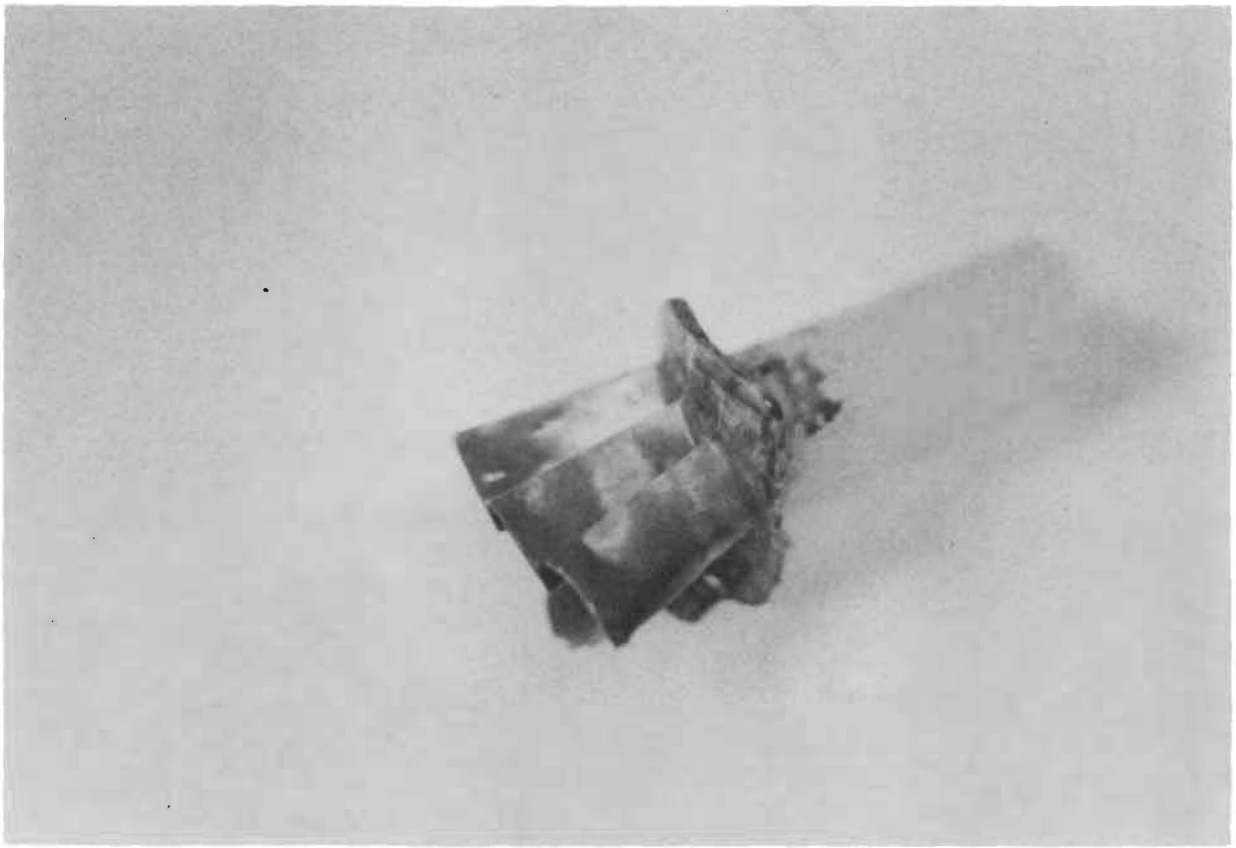

Figure 2. Wax Pattern Dipped in Carbon Slurry and Allowed to Drain for Thirty Seconds.

bubbles resulting in the same bubbly surface as was present without vacuum evacuation.

6. Dewaxing

Two methods of removing the wax from the dry carbon molds were used. One involved the use of microwave energy to melt the wax, the other used a hot iron and low temperature oven to melt out the wax. Both methods were followed by a chemical cleaning.

In the hot iron technique the majority of the large cylinder of wax in the center of the mold was "cut out" with the hot iron. This localized heating did not cause enough expansion of the wax to crack the mold. The mold was then placed in an oven at 100°C to melt out the remainder of the wax. Again the mold did not crack due to the absence of the large volume of wax in the center of the mold. After about two hours in the oven the mold was removed and rinsed two or three times with toluene heated to about 100°C to dissolve the remaining wax film. Each rinse was left in the mold for about three minutes. The mold was then rinsed with acetone at room temperature to remove the toluene and placed in a 100°C oven to evaporate the acetone. Microwave dewaxing was done by placing the mold in a metal chamber and bombarding it with microwave energy. A water load of 200 ml was also put in the chamber. About 800 watts (200 watts of which were reflected) was fed into the chamber for one minute. The wax melted at the surface of the mold only, providing a cushion which prevented subsequent thermal expansion of the wax from cracking the mold. The mold was placed immediately in a 100°C oven to melt out the rest of the wax before the

cushion of molten wax at the surface resolidified, and then cleaned chemically as described above. Finished molds ready for filling are illustrated in Figure 3.

During the meltout and subsequent chemical cleaning of the mold the wax permeated the mold clogging the pores. The last two toluene rinses removed a portion of this wax, but some remained in the pores of the mold. This made casting in the mold slow and more difficult.

C. Casting into the Molds

In order to check the surface quality available in the molds a full rotor and 3 three-blade rotor section molds were filled with fused silica slip. The slip was placed under vacuum to remove air and filling was also done under vacuum. After filling, the molds were allowed to air dry for about two days, then dried in a 100°C oven for 12 hours. These test pieces were fired to 1200°C in air, cooled, and examined. The fired pieces were quite weak and easily broken, but examination of the blade surface was possible. One three-blade section had a sufficiently smooth surface, but the other sections and the full rotor were rough and grainy as can be seen in Figure 4. The extremely long casting time due to the wax clogged molds may have allowed the PVA binder to partially dissolve into the slip, leaving a rough surface.

A silicon carbide slip was prepared according to instructions received from Ford. This slip was cast into two, three-blade section molds using a vibrator to promote flow and filling of the mold. No vacuum was used. The molds were allowed to dry at room temperature for 12 hours then placed in a 100°C oven for 12 hours to complete drying. After drying a large amount of shrinkage which can be seen in Figure 5 had occured causing small

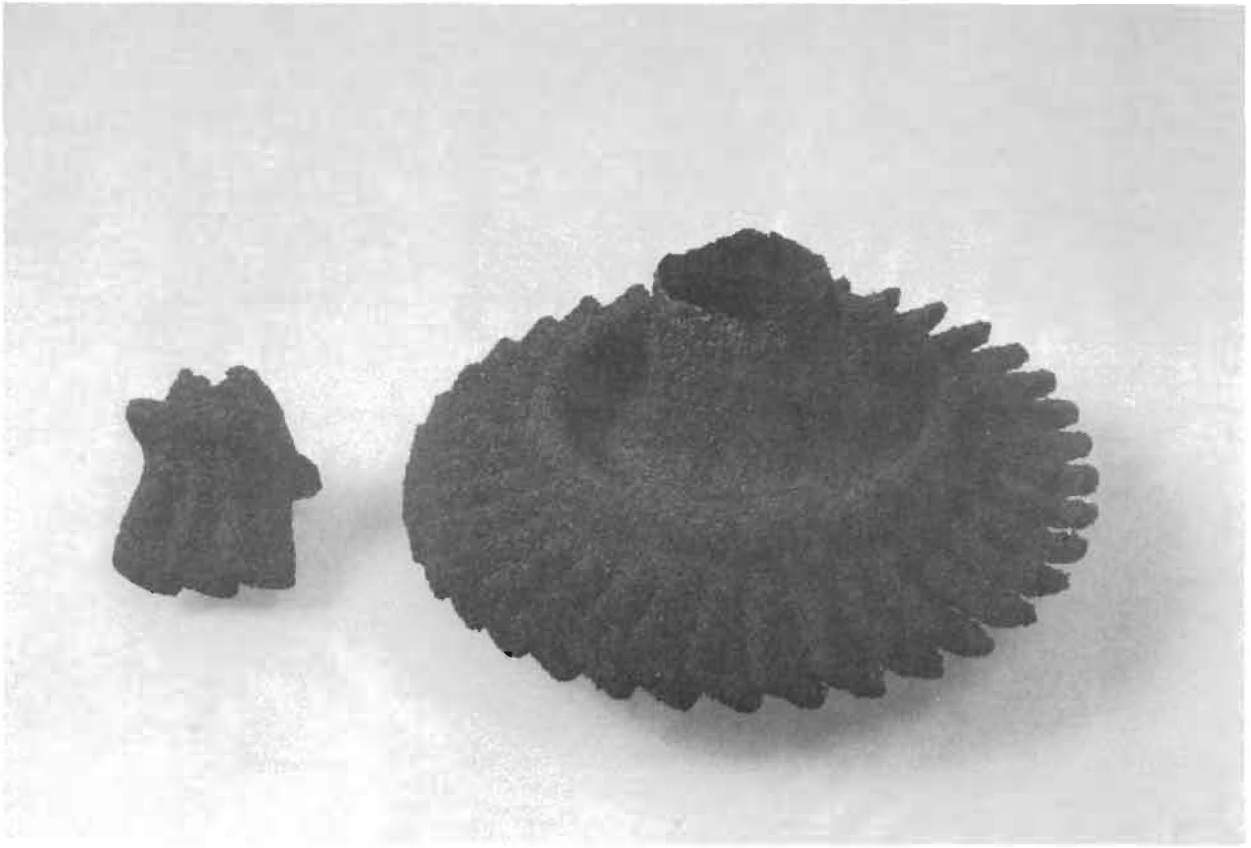

Figure 3. Finished, Dewaxed Molds Ready for Filling with Silicon Carbide Slip.

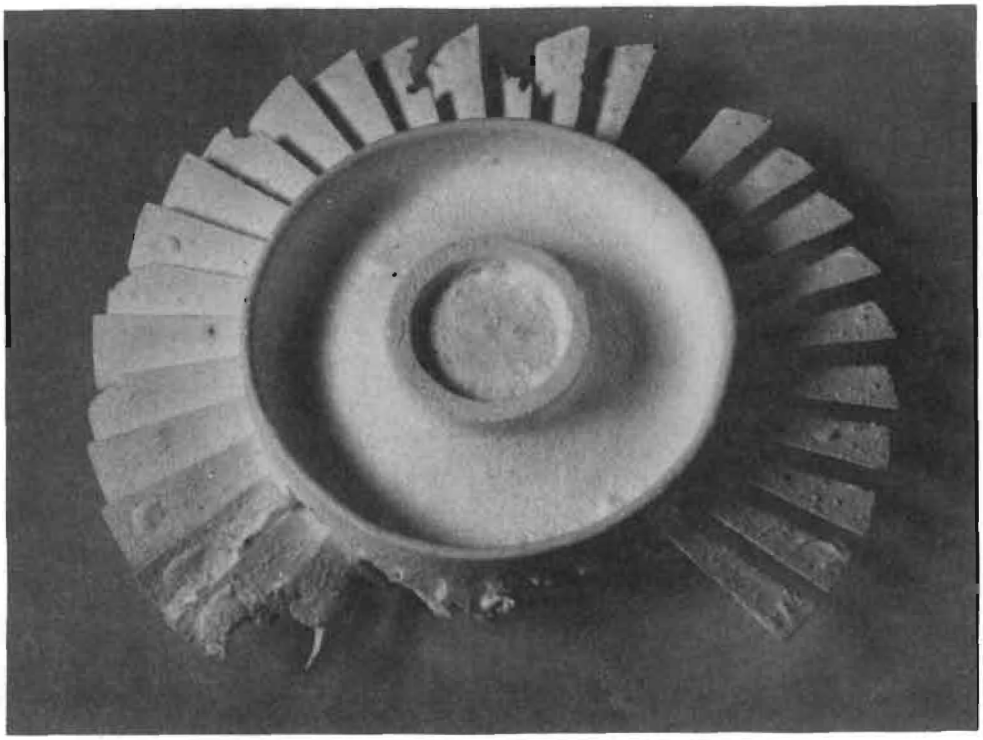

Figure 4. Fused Silica Test Casting Showing Rough Surface Obtained.

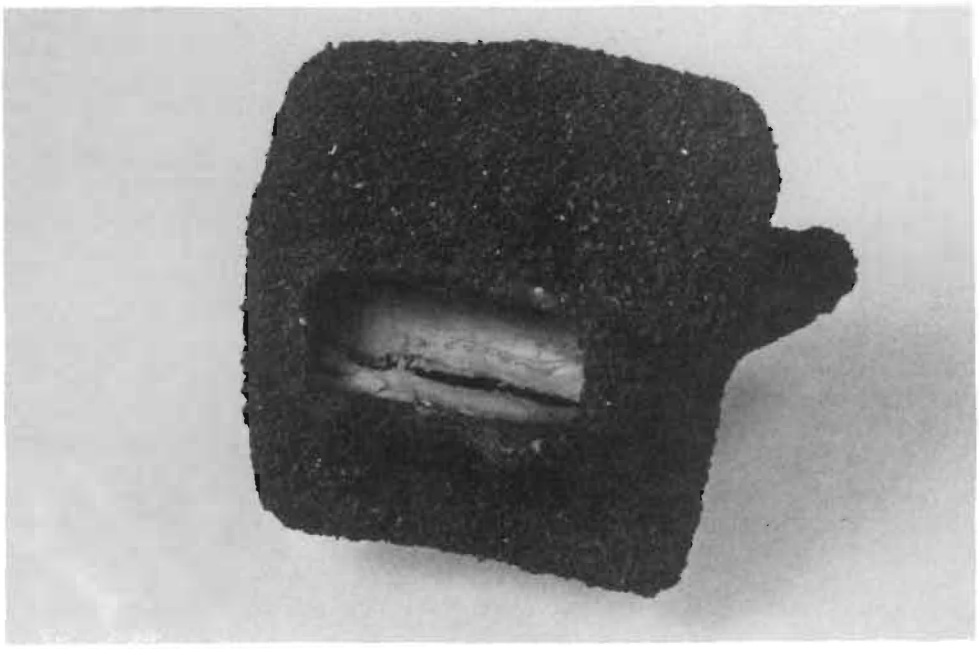

Figure 5. Three-Blade Section Carbon Mold Filled with Silicon Carbide Slip.

cracks in one of the sections. In a larger piece, such as a full rotor, this shrinkage could not be tolerated unless the molds were removed before drying proceeded beyond a "safe" point.

D. Sintering of Silicon Carbide

The uncracked three-blade section and mold were placed in a vacuum furnace equipped with a carbon resistance heating element. Pressure was reduced to and held below $10^{-3}$ torr for the sintering process. Temperature was measured by sighting onto the sample with a micro optical pyrometer, and heating was carried out according to the schedule provided by Ford. After a short time at sintering temperature, the furnace failed and rapidly cooled. The mold had disintegrated and was washed off the three-blade section with running water. The silicon carbide section was cracked and weak, but sintering had started and the surface was smooth. The mold and silicon carbide section are shown in Figure 6.

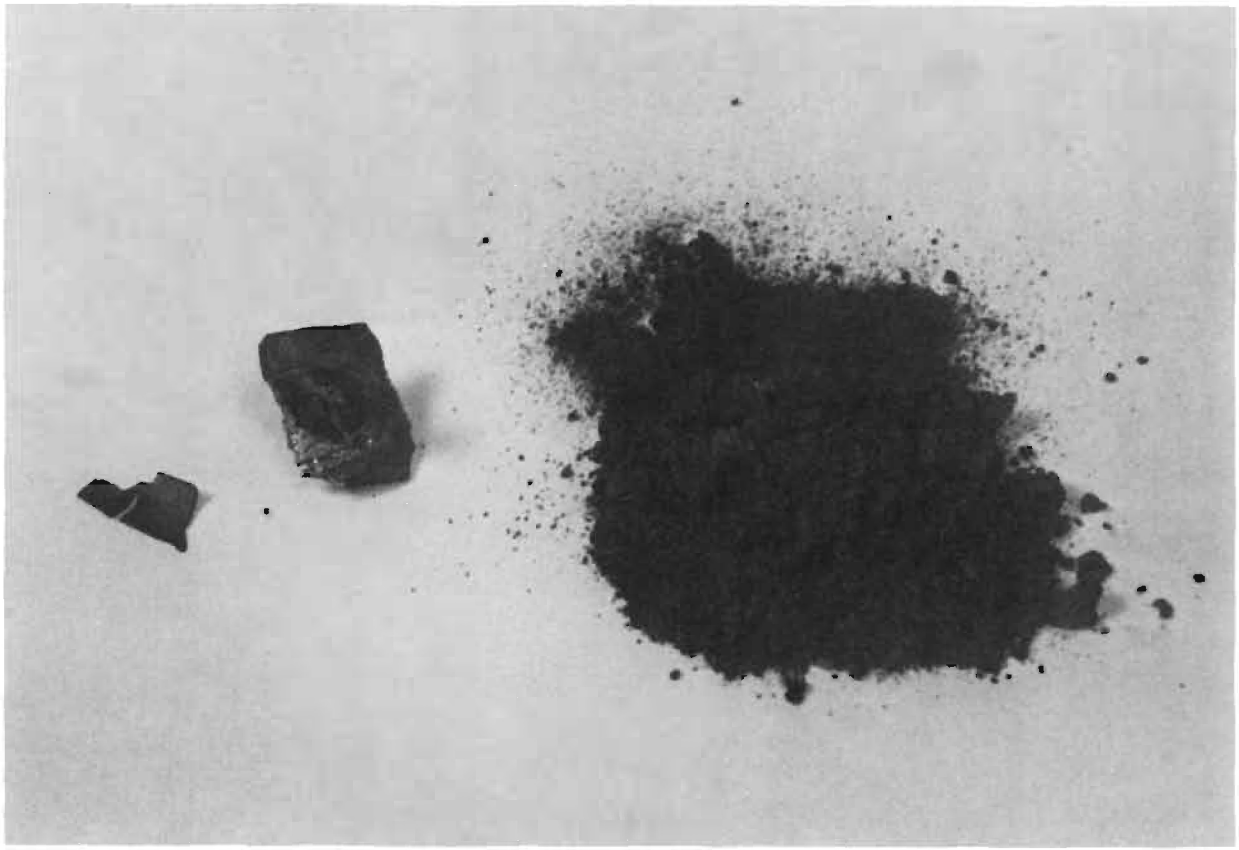

Figure 6. Sintered Silicon Carbide Three-Blade Section, Broken Blade, and Disintegrated Carbon Mold After Vacuum Firing.

## IV. DISCUSSION OF RESULTS

In the dipping process used to build up the molds no satisfactory wetting agent was found that would work with solvent soluble wax, water soluble binder system that was employed. Stearates were useful in wetting the wax without dissolving it, but dissolved out during dewaxing leaving a rough surface. Other wetting agents that are water soluble would not wet the wax. Although it has been shown that the carbon shell mold process is feasible for the production of silicon carbide parts it is now known that a modification or change in the particular system used for this evaluation will have to be made to insure a smooth surfaced, easily slip cast part. Several possibilities are discussed in the recommendations section of this report.

The molds produced had insufficient porosity for rapid slip casting with a water suspended slip. The dewaxing process dispersed wax throughout the mold closing the pores. It was noted, however, that the mold was permeable to some solvents, such as toluene and trichlorethylene, as they dissolved the wax, opening the pores. This behavior suggests the possibility of casting solvent suspended slip into the molds.

The polyvinyl alcohol (PVA) binder was not affected by hot toluene or trichloroethylene even over extended periods of time, but it was quickly dissolved by water even after completely drying. This action helps to explain the rough surfaces obtained on parts cast with water suspended slips. The water dissolved out part of the mold causing the carbon particles to pull out and create a rough surface.

The molds disintegrated completely during heating in a vacuum as the PVA carbonized and no binder remained leaving a carbon powder mold. This would also occur in an inert gas atmosphere. The mold disintegration occured early in the firing cycle so that shrinkage of the part during sintering was not impeded by the mold. Shrinkage of the silicon carbide slip during the drying process would have to be controlled on a large piece to prevent cracking. The PVA bond softened at about 150$^{o}$C suggesting the possibility of drying the mold and part at this temperature to allow the part to shrink without cracking.

The silicon carbide three-blade section that was sintered demonstrated that the molds could be used as they are now being made for the production of these parts. The section did crack and the blades were easily broken off, but this is attributed to the furnace failure and extremely rapid cool down. Also, certain metal parts of the furnace melted and dripped onto the part being sintered. This obviously contributed to the poor condition of the part. The most important facts to be gained by the firing of the part are that it did sinter and produce a three-blade section, and that the mold did disintegrate leaving the part.

## V. PROJECT ACCOMPLISHMENTS

A method of producing organically bound carbon molds has been developed. The method lends itself to industrial production line techniques.

The feasibility of using these molds for investment casting has been demonstrated. The molds disintegrate upon heating in inert gas or vacuum and burn in air.

A small silicon carbide part, a three-blade rotor section, has been sintered in one of the carbon molds. The disintegrated mold was easily removed and the part remained.

# VI. RECOMMENDATIONS

## A. General Discussion of Mold Fabrication

Two problems were encountered with the polyvinyl alcohol bound carbon molds discussed in this report. One was the difficulty in obtaining a smooth uniform surface because of the inability of the carbon-PVA slurry to wet the wax surface of the pattern. The other was that the mold's porosity was reduced or closed off completely by the wax during dewaxing. Several possible solutions to these problems that should be investigated are outlined below.

A wetting agent, Eastman "Aerosol OT," has been suggested by Yates Manufacturing Company, Chicago as a means of wetting the wax surface. This agent is mixed with water and sprayed onto the surface of the pattern leaving a very thin deposit. This deposit is wet by water. When the mold is dewaxed, the deposit will possibly wash out, but the thinness of the layer may leave a smooth surface despite the washing out.

A completely different system of binder and wax may be the ultimate solution to the problems. Use of a water soluble wax and a solvent dispersed carbon slurry with a suitable binder would allow the slip itself to further dissolve wax clogging mold pores. Yates Manufacturing indicates that their water soluble wax dissolves quickly and spontaneously in water so little or no clogging should occur. Another method of obtaining the same result would be to use the present system and cast a solvent dispersed slip into the mold to dissolve the wax out of the pores as casting progressed.

Another possibility is the use of a cured plastic mold made from epoxy, polyester, phenolic, or other resin. A variety of properties are available in these plastics and all will carbonize and disintegrate when heated, provided

their structure is not cross-linked during curing. An epoxy is available that will withstand continuous temperatures of 250°C and intermittent temperatures up to 350°C. These materials are said to be porous when 10-15 percent filler, in this case carbon, is added before curing. Wetting of the wax pattern would not be required as the pattern would be submerged in the liquid plastic during curing. The wax could be removed by "smoking out" at 400-600°F or is it were water soluble by rinsing the cured plastic mold in water.

A solution of camphor in naphthalene has been used as a waxy glue in vacuum systems. This substance sublimes as pressure drops leaving no residue. A pattern made from an appropriate solution could be evaporated out of the mold, leaving an unclogged porous mold. The wettability or solubility of this substance is questionable and would have to be determined.

B. Silicon Carbide Slip

Slip has been prepared according to Ford's instructions using the General Electric powders furnished. This slip has been cast in a carbon mold and exhibited considerable shrinkage on drying. The amount of shrinkage would probably prohibit the casting of a larger piece, such as a full turbine rotor, indicating a modification of the slip is needed. Reduction of liquid content, suspension in an organic solvent, modification of particle size distribution, or a combination of modifications could act to reduce the large amount of shrinkage. The mold in which the slip is to be used would partially determine the modifications that need to be made. Conversely, if a slip of better characteristics could be developed this would dictate the mold that would be developed and used.